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Polyvinyl chloride microplastic detection by single particle inductively coupled plasma mass spectrometry for the characterization of model microplastics

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Methodology based on the use of single particle inductively coupled plasma mass spectrometry (SP-ICP-MS) for the detection and quantification of polyvinyl chloride (PVC) micro/nanoplastics was developed. The use of chlorine as an intrinsic element present in the composition of the polymer improved the performance of SP-ICP-MS reported in the literature, allowing the detection of PVC particles down to 0.35 μm, by monitoring the ^{35}Cl isotope using a quadrupole-based instrument. A top-down protocol for preparation of microplastic suspensions based on cryogenic grinding of the raw plastic material in combination with fractionation by centrifugation was proposed, to be used as a model material in risk assessment studies. The model suspensions were characterized by SP-ICP-MS and dynamic image analysis (DIA). The size of the PVC particles was in the range of 0.4–5.7 μm, with a median equivalent diameter of 1.0 μm, whereas the concentration of the suspensions was in the range of 10^9 L^{-1}, both determined by SP-ICP-MS. The particles showed an irregular shape confirmed by DIA, with a mean aspect ratio of 0.67 and circularity of 0.79. The SP-ICP-MS method developed was also applied as a proof of concept to the screening of PVC microplastics released from industrial products.

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1. Introduction

Global concern about issues derived from the use of plastics is well known. Because of their properties and versatility, plastics have acquired a fundamental role in our daily lives and in strategic sectors such as the transport, construction, electronics, healthcare, textile and food industries.[1,2] Despite their durability, plastics can undergo degradation processes through different mechanisms, including mechanical erosion, photodegradation, chemical corrosion, biological degradation or thermal fragmentation.[3] Once they are released into the environment, they suffer from different transformation and degradation processes that can lead to the formation of microplastics and nanoplastics. Whereas microplastics are defined as small particles of plastic typically no more than 5 mm across, there is still no consensus on the definition of nanoplastics.[4,5] Some authors establish the nanoplastic threshold at 1 μm, others place the nanoplastic upper size limit at 100 nm. However, definition of a nanoplastic as a particle measuring no more than 1 μm across in any one dimension is widely accepted as a guiding definition.[6]

Although there is currently no regulation in place applying to microplastics in a comprehensive manner, the European Commission adopted in 2023 a REACH restriction on microplastics intentionally added to products and a proposal for regulation on preventing plastic pellet losses to the environment.[7,8] However, the presence of micro/nanoplastics is not only limited to the environment, as they are present in different consumer products, cosmetics or food, leading to increasing exposure for human populations and hence, risk to human health.[9,10] Recent studies reporting the detection of microplastics within the human body have raised concern among the scientific community, authorities, politicians and general public, as the knowledge about human exposure is still very limited and their impact on health is still not well understood.[11] Their small size but large surface area promotes their chemical interactions with physiological fluids and tissues.[12]

Although there are a number of important existing techniques for the detection of plastic particles, mostly based on the use of microscopy, Fourier-transform infrared, Raman spectroscopy in combination with microscopy and imaging analysis (micro-FTIR and micro-Raman, respectively) or pyrolysis and thermoextraction and desorption gas chromatography mass spectrometry (Py-GC-MS and TED-GC-MS, respectively),[12,13] quantification and characterization of micro/nanoplastics remains a controversial issue, and limitations come into play

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for detection, characterization and quantification of the smallest particles. In fact, it is thought that the smallest particles may pose the greatest risk, as they are probably the most numerous, have a higher reactivity due to their higher surface area and can cross biological barriers more easily. Analytical techniques widely used to identify microplastics are size dependent, allowing for instance the detection of particles with diameters >1 μm in the case of Raman spectroscopy or even >5–10 μm when using μ-FTIR techniques.[13,14] Besides all these techniques, dynamic image analysis (DIA) has also been shown to be an effective technique for quick and accurate characterization of particles from 2 μm to 300 μm, being able to provide not just size distributions, but also morphological information about their shape.[15,16] In recent years, the applicability of single particles (SP-ICP-MS) for the detection of microplastics has been reported by monitoring carbon isotopes (^{12}C, ^{13}C),[17,18] as this element is their main component, providing information on both the number concentration and size distribution (when information about the nature of the plastics is known) of microplastics in real samples. However, the poor performance of this element in ICP-MS establishes size detection limits of around 1 μm. For this reason, the monitoring of other elements present in the plastic matrix could be a useful alternative in the detection of nanoplastics. In this sense, although the determination of halogens by ICP-MS could be challenging,[19] fluorine has been successfully used for the detection of fluoropolymers, such as polytetrafluoroethylene (PTFE),[20–22] with size detection limits of PTFE particles of around 1 μm. Chlorine has also been reported in the analysis of polyvinyl chloride (PVC) materials by time-of-flight mass analyzers (ICP-TOF-MS), with critical size values of 3.9 μm or 1.3 μm, when monitoring $^{35}Cl^+$ or $^{35}ClH_2^+$, respectively.[23,24]

Therefore, the development of analytical methods for the correct identification, characterization and quantification of micro/nanoplastics is essential to carry out an adequate risk assessment of the possible harmful and toxicological effects of these materials on health. However, the lack of different types of model microplastics is an important drawback, as in practice commercially available standards are mainly restricted to polystyrene (PS) and polyethylene (PE) particles of different diameters.[25] In contrast, polypropylene (PP), PVC, polyurethane (PU), and polyethylene terephthalate (PET), which constitute an important fraction of the total plastic production, are harder to obtain. Consequently, most current studies about the potential human toxicity and ecotoxicity have been conducted on polystyrene spheres,[26–28] even if this kind of polymer only accounts for about 5% of total global plastic production.[29] For this reason, the need to develop model materials that can be used as a starting point for the development and optimization of new methodologies for the detection of real microplastics of different nature, shape and size is of great interest and importance to help elucidate the potential risk and toxicity of micro/nanoplastics.

The aim of this work is the development of a methodological platform based on the use of single particle ICP-MS (SP-ICP-MS) and dynamic image analysis (DIA) for the determination of number concentrations and the morphological characterization (size and shape) of PVC microparticle suspensions. Although both DIA and SP-ICP-MS can provide information on number concentration by counting particles in the analyzed suspension, the two techniques complement each other. Although DIA lacks selectivity in terms of particle detection, it provides direct information on size and shape, whereas detection in SP-ICP-MS is based on the element monitored, and information on size is derived from the mass of the element in the particle once its composition and shape are known or assumed. Preparation protocols of PVC microplastic suspensions based on cryogenic grinding of the raw plastic material, in combination with fractionation by centrifugation and subsequent analysis by SP-ICP-MS and DIA for their characterization, have been proposed. The use of chlorine as an intrinsic label of PVC has been explored for the detection of these micro/nanoparticles to improve size detection limits by SP-ICP-MS. This methodology has also been applied to the detection of PVC micro/nanoparticles released from industrial products as a proof of concept.

2. Experimental

2.1. Instrumentation

A PerkinElmer NexION 2000B Inductively Coupled Plasma Mass Spectrometer (Toronto, Canada) was used throughout the study. The sample introduction system consisted of an Asperon™ linear pass spray chamber (PerkinElmer, Toronto, Canada), equipped with a flow focusing nebulizer (Ingeniatrics, Sevilla, Spain). A μDx Single Cell Autosampler (Elemental Scientific, Omaha, NE, USA) equipped with a syringe pump operating at 10 μL min^{-1} was used for sample introduction. Default instrumental and data acquisition parameters are listed in Table 1. Data were acquired in single particle mode using the Syngistix Nano-application module version 3.2 (PerkinElmer Inc). Recorded scan files were exported and processed with the SPCal[30] software version 1.3.3 using iterative thresholding and the Poisson ($\alpha = 2.867 \times 10^{-7}$; Curie expression, $\eta = 1$, $\varepsilon = 1$) or the Gaussian ($\alpha = 2.867 \times 10^{-7}$; 5σ) filter option, depending on the baseline intensity.[31] Since exported data from Syngistix

Table 1 Default instrumental and data acquisition parameters for SP-ICP-MS

Instrumental parameters	
RF power	1600 W
Argon gas flow rate	
Plasma	15 L min^{-1}
Auxiliary	1.2 L min^{-1}
Nebulizer	1.0 L min^{-1}
Make-up	0.2 L min^{-1}
Sample flow rate	10 μL min^{-1}
Data acquisition parameters	
Dwell time	200 μs
Total acquisition time	60/180 s
Isotopes monitored	^{13}C and ^{35}Cl

consisted of a real number (*e.g.*, 23.000019) they were truncated to the corresponding integers before processing with SPCal. The particle size method was used for the calculation of dissolved element transport efficiency.[32,33] For this purpose, reference latex sphere suspensions (RM165) of 2.223 ± 0.013 μm diameter obtained from BCR and aqueous carbon solutions were used, obtaining a transport efficiency value of 76%. Equivalent diameters were further calculated from the mass of element per particle determined from the calibration of dissolved carbon or chlorine standards.[32] Particle number concentration was determined against a calibration prepared from RM165 (3.35×10^{10} L^{-1}).

An XPT-C Particle Analyzer detector (PS Prozesstechnik GmbH, Suiza), equipped with a CCD camera that allows live image capture, was used for dynamic image analysis. Measurement conditions for size determination were: threshold 98, sharpness 200, noise 0; whereas threshold 170, sharpness 1500, noise 2 were used for the determination of the number of particles. A light intensity of 528 (arbitrary units) was used in both cases.

A JXA-iHP200F Field Emission Electron Probe Microanalyzer (FE-EPMA) equipped with four wavelength dispersive spectroscopy (WDS) detectors was also used for visualization of the PVC particles in the final suspension and its elemental analysis.

2.2. Standards

Aqueous carbon and chloride solutions were prepared from commercially available standard stock solutions of 1000 mg L^{-1} prepared from tartaric acid in 0.2% HNO_3 (*v*/*v*) and ammonium chloride in water, respectively (Inorganic Ventures, Christiansburg, VA) by dilution in ultrapure water (Milli-Q Advantage, Molsheim, France). Reference latex sphere suspensions of 2.223 ± 0.013 μm diameter (RM165) were obtained from BCR (Geel, Belgium) and used for the calculation of nebulization efficiency.[32,33] Dilutions were prepared in ultrapure water and glass vials (Labbox, Spain) by accurately weighing (±0.1 mg) aliquots of the stock suspensions after 1 min of sonication (Bandelin Sonorex™ Super RK31, Bandelin, Germany).

2.3. Materials and samples

Commercial polyvinyl chloride (PVC) powder (Sigma, St. Louis, MO, USA) was used as a model sample. PVC food-grade hoses (5 m long, 10 mm diameter) and unplasticized PVC pipes (15 mm diameter) intended for pressurized water and sewage networks were purchased from a department store and used in migration assays.

2.4. Procedures

2.4.1 Preparation of model microplastic suspensions. For the preparation of model microplastic suspensions, the commercial PVC powder described in Section 2.3 was used and subjected to a cryogenic grinding process with liquid nitrogen to reduce the size of the material. An MM400 ball mill (Retsch, Haan, Germany) and stainless-steel containers were used for this purpose. Before each milling, containers filled with the raw material were placed in a liquid nitrogen bath for 5 min. Two cycles of 150 s each at 30 Hz were performed to mill 5 g of PVC (2.5 g in each container). Frequency and times were adjusted following the recommendations of the mill manufacturer, whereas the number of cycles was optimized by visual inspection after each cycle, in terms of homogeneity and particle size. Once ground, 0.17 g of the PVC material was weighed and suspended in 17 mL of ultrapure water using glass vials. After 1 min of sonication at room temperature in an ultrasonic bath sonicator (Bandelin Sonorex™ Super RK31, Bandelin, Germany), 5 mL fractions were taken and centrifuged at 120 g for 3.60 min and 20 °C (Heraeus Multifuge X1R, Thermo Fisher Scientific, USA). Centrifugation conditions were set considering the density of the PVC material (ρ: 1.38 g cm^{-3}) to remove particles larger than 3 μm, while keeping smaller micro/nanoplastic particles in the supernatant. Once the supernatant containing particles <3 μm was obtained, suspensions could be analyzed by SP-ICP-MS and DIA.

2.4.2 Determination of size distributions by laser diffraction. A Mastersizer 3000E (Malvern Panalytical, UK) was used to characterize the size distribution of the PVC material before and after grinding. Less than 1 g was introduced as a solid dispersion by aspiration. Five replicates were made for each sample.

2.4.3 Migration of PVC microplastics from industrial products. PVC food-grade hoses were cut into 50 cm length (internal volume of 8–9 mL) and filled with ultrapure water and/or 3% (w/v) acetic acid (food simulant). For the migration assays with ultrapure water, two different temperatures (20 °C and 70 °C) and two times (24 h and 72 h) were tested. On the other hand, following the procedure established by regulation no. 10/2011 of the European Commission[34] on plastic materials and articles intended to come into contact with food, a microplastic migration study was performed with 3% acetic acid for 10 days at 40 °C in an incubator (OVAN, Barcelona, Spain). This regulation establishes specific requirements for the manufacture and marketing of plastic materials and articles that are designed to come into contact with food, are already in contact with food or can reasonably be expected to come into contact with food. The working temperature range recommended by the manufacturer for this kind of food-grade hose was −20 °C to 60 °C.

Unplasticized PVC pipes used to carry pressurized water (drain tubes) were cut into 6 cm length fragments (internal volume of 11–12 mL) and filled with ultrapure water. Migration assays were performed at room temperature (20 °C), 40 °C and 60 °C for 72 h, taking into account that the maximum working temperature recommended by the manufacturer is 45 °C in continuous operation, restricting the use at higher temperatures to occasional discharges.

3. Results and discussion

3.1. Size reduction of the raw PVC material

One of the main limitations in the development of a comprehensive risk assessment of micro/nanoplastics lies in the lack of model microplastic materials of different compositions. Among the different types of plastics, polyvinyl chloride (PVC) is one of the most widely used by the industry. Given its frequent use,

there is a need for well-characterized PVC microplastic suspensions that can be used as model or control materials in risk assessment assays. For this purpose, the original PVC raw material was first cryogenically ground and size reduction was confirmed by laser diffraction. Size distributions obtained by laser diffraction (Fig. S1) showed that the cryogenic grinding process led to an effective reduction of the material size, going from around 2% to 30% of volume population below 100 μm for the original raw and ground material, respectively. In addition, a second population of smaller particles with a maximum below 30 μm was observed for the PVC cryogenically ground material, with <2% of volume population below 10 μm. Particles below 10 μm were not observed in the original raw material. Consequently, PVC model microplastic suspensions were prepared from the cryogenically ground material, with the aim of using them in the development of methodologies based on SP-ICP-MS for the characterization of PVC microplastics.

3.2. Preparation of PVC model suspensions: isolation of the particle fraction of interest

Since the particle size range of interest and optimal size working range for SP-ICP-MS are up to 3–5 μm,[18,35] a sample fractionation procedure was performed by centrifugation, taking into account the PVC density and a desired upper size limit of 3 μm, assuming spherical particles (Section 2.4). As a first approach, suspensions resulting from these assays were analyzed by DIA. Particle number concentration of $(3.35 \pm 0.14) \times 10^8$ L^{-1} ($n = 3$) was obtained for PVC <3 μm suspensions. Fig. 1 shows the size distribution obtained by DIA for the nominal fraction of PVC particles <3 μm. Equivalent diameters of (2.98 ± 0.09) μm were obtained by this technique, although particles larger than 3 μm (established upper limit in the fractionation step) were also detected, which would be justified by the fact that spherical particles were assumed for the cut-off calculations during centrifugation. However, actual particles in the suspension were shown to have irregular shapes (Fig. S2), which would explain the detection of larger particles by DIA. In this sense, different parameters[36] such as bounding rectangle long side (4.00 ± 0.14 μm), bounding rectangle short side (2.54 ± 0.09 μm), aspect ratio (0.67 ± 0.01 μm) and/or circularity (0.79 ± 0.01 μm) were considered to confirm that PVC particles in model micro/nanoplastic suspensions were not spherical. Additional techniques such as Field Emission Electron Probe Microanalyzer (FE-EPMA) and wavelength dispersive spectroscopy (WDS) also confirmed the presence of non-spherical particles whose composition was associated with carbon and chlorine (Fig. S3).

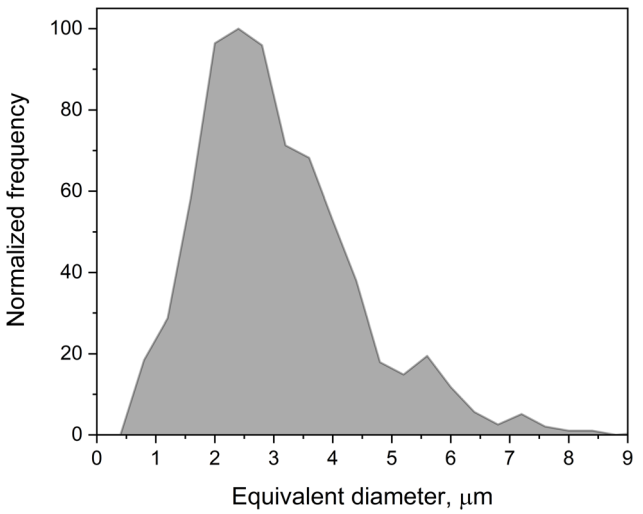

Fig. 1 Size distribution histogram obtained by DIA for PVC fraction <3 μm suspension (dilution 1 : 3).

3.3. Characterization of PVC model suspensions by SP-ICP-MS

After the preparation of PVC model microplastic suspensions by fractionation to sizes <3 μm their detection and characterization by SP-ICP-MS was carried out. The use of SP-ICP-MS for the detection of microplastics has been described in the literature mainly through the monitoring of carbon, the main constituent element of polymers. However, the poor performance of this element in ICP-MS (low sensitivity and high background levels) makes it necessary to search for alternatives that allow the detection of micro/nanoplastics. Laborda *et al.*[18] achieved the detection of polystyrene microparticles down to 1.2 μm by monitoring the ^{13}C isotope. Hendriks *et al.*[37] obtained a size limit of detection of 1.56 μm, comparable to values reported by Harycki *et al.*[38] (1.8 μm), both using ICP-TOF-MS instrumentation. Likewise, strategies based on the labelling of micro/nanoplastics with metals have been proposed in the literature.[39]

In this work, the use of intrinsic labels from the material itself is proposed, which would allow achieving lower size detection limits than those reported in the bibliography. Thus, in the case of PVC particles, chlorine monitoring through the ^{35}Cl isotope is proposed, since this element is present in the monomer structure (–[CH_2–CHCl–]$_n$). However, ^{13}C was also monitored for control and comparative purposes. The ^{37}Cl isotope was discarded due to detector saturation coming from $^1H^{36}Ar$ interference. Likewise, the use of He and NH_3 as collision/reaction cell gases was explored to reduce $^{16}O^{18}O^1H^+$ interference in $m/z = 35$, but no improvements in detection limits were observed in comparison to the "no gas" mode, as other authors have shown.[40,41] Table 2 shows SP-ICP-MS results for the analysis of the PVC <3 μm suspension. Fig. 2 shows a comparison of the size distribution histograms obtained by SP-ICP-MS for ^{35}Cl and ^{13}C. Sizes in Table 2 and Fig. 2 are reported as equivalent diameters assuming spherical shapes and considering the chemical composition and density of the PVC microparticles detected (chlorine mass fraction in PVC particle, $\chi_{Cl} = 0.567$; carbon mass fraction in PVC particle, $\chi_C = 0.384$; $\rho = 1.38$ g cm^{-3}).

As can be seen from Table 2 and Fig. 2, monitoring the ^{35}Cl isotope significantly improved the particle size detection limit in comparison to ^{13}C monitoring, going from 1.07 μm for ^{13}C to 0.35 μm in the case of ^{35}Cl. This value also improves on the ones reported in the literature when monitoring chlorine by ICP-TOF-MS.[23,24] As a result, a higher number of particles in a wider

Table 2 SP-ICP-MS results obtained for the PVC <3 μm suspension. Mean ± standard deviation ($n = 5$)

Suspension	Element	Mean baseline intensity, counts	Particle number concentration, L^{-1}	Size range[a], μm
Fraction <3 μm	^{35}Cl	3.40 ± 0.06	$(1.63 \pm 0.07) \times 10^9$	0.37–5.70 (1.04)
	^{13}C	4.58 ± 0.02	$(3.33 \pm 0.06) \times 10^8$	1.30–4.23 (1.99)

[a] Median size value in brackets.

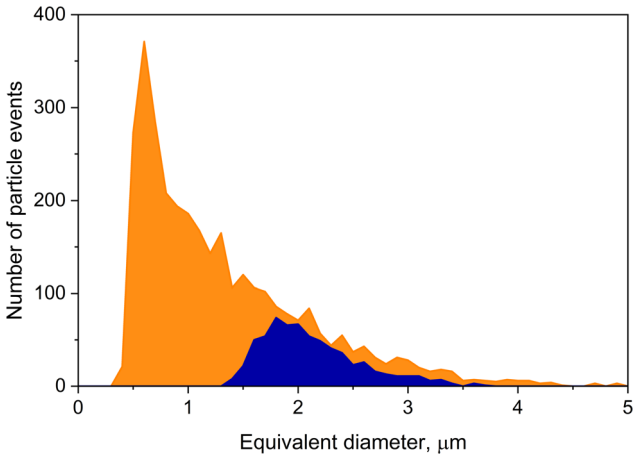

Fig. 2 Comparison of size distribution histograms obtained by SP-ICP-MS for the PVC <3 μm suspension (orange: ^{35}Cl; blue: ^{13}C) (dilution 1 : 3).

range of sizes were detected when monitoring chlorine. Thus, only particles larger than 1 μm (mainly around 2 μm) were detected when monitoring ^{13}C, which is consistent with results previously obtained by DIA (Fig. 1) and values obtained from the literature. Particle number concentration calculated for ^{13}C by SP-ICP-MS also agreed with the one previously obtained by DIA ($(3.35 \pm 0.14) \times 10^8$ L^{-1}), as monitoring of carbon and the DIA technique have similar size limits in the low range. On the other hand, particles down to 0.37 μm were detected when monitoring ^{35}Cl, improving the SP-ICP-MS micro/nanoparticle detection capability. However, it should be noted that particles over 3 μm could be underestimated because of their lower nebulization efficiency. Thus, the results shown here confirm the presence of PVC particles in the hundreds of nanometre range, allowing the size detection limit usually obtained in the analysis of microplastics to be decreased.

3.4. Detection of PVC microplastics released from industrial products

Once the feasibility of SP-ICP-MS for the detection and characterization of PVC microparticles by monitoring ^{35}Cl was demonstrated, the analysis of samples that could potentially contain this kind of microplastics was addressed. The use of certain plastic materials in different kinds of hoses or pipes is very common in a wide variety of industrial sectors, such as the food industry, as they allow the circulation of raw materials throughout the production process. Therefore, and given the global concern about the possible presence of micro/nanoplastics in the food and environment, the release of PVC microparticles from PVC food-grade hoses or/and unplasticized PVC pipes used to carry pressurized water (evacuation tubes) was studied.

3.4.1 Release of PVC microplastics from food-grade hoses. In the case of PVC food-grade hoses, migration assays were firstly performed in ultrapure water at different times and working temperatures (Section 2.4), both within and outside the recommended temperature range specified by the supplier. Fig. 3 shows the time scans obtained for the migration assays performed at 20 °C and 70 °C. Time scan for ultrapure water is shown in Fig. S4 for comparative purposes. In order to discriminate particle events from the baseline, critical values (Y_c)[42,43] were estimated using a Gaussian filter ($\alpha = 2.867 \times 10^{-7}$; 5σ) in the case of baselines over 10 counts or a Poisson one for baselines below 10 counts ($\alpha = 2.867 \times 10^{-7}$; Curie expression; $\eta = 1$; $\varepsilon = 1$).[31]

Once a critical value for discrimination of particle events from the baseline is applied, the assessment of the presence or absence of particles in a sample relies on the quality of the available blanks. Under ideal conditions, no particles would be detected in the blank samples and hence the recording of one event higher than the signal intensity critical value Y_C in the time scan would confirm the presence of particles in the sample. However, when particle events are detected in the blanks, eqn (1) must be used for estimation of the critical value for particle detection ($Y_{C,\,N}$):

$$Y_{C,N} = Y_{B,N} + 2.33\sigma_{B,N} = Y_{B,N} + 2.33\sqrt{Y_{B,N}} \quad (1)$$

where $Y_{C,\,N}$ is the critical value for the number of events, $Y_{B,\,N}$ the mean number of particle events in the blank and $\sigma_{B,\,N}$ its standard deviation.

Therefore, after applying the threshold criterion for identification of particle events in the time scan, the occurrence of particle events in ultrapure water was around 21 events, meaning a critical value for particle detection of $Y_{C,\,N} = 32$ events after the application of eqn (1), which would correspond to a particle number concentration critical value (X_C^{number}) of 1.8×10^6 L^{-1}. Fig. 4 shows that no particles were detected in the migration assays performed at 20 °C for 24 h and 72 h, as well as at 70 °C for 24 h, although chlorine was detected in the baseline, which would be related to the presence of dissolved chlorine-containing species or particles below the size detection limit released. In all these cases, the number of events detected was very low, with values between 2 and 4 events, which are below the critical value calculated for ultrapure water under the measurement conditions. On the other hand, for the migration assay carried out at 70 °C for 72 h, a significant number of

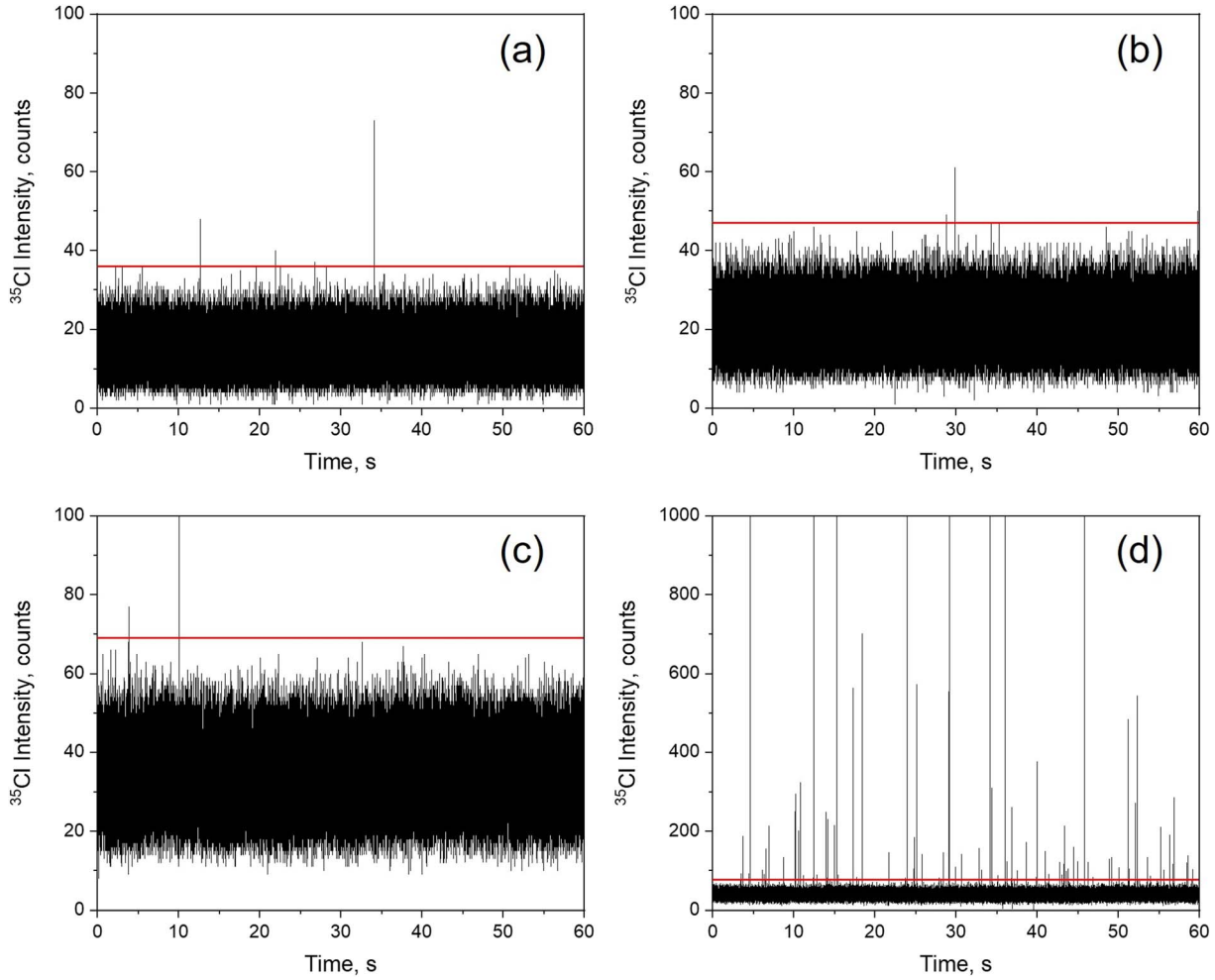

Fig. 3 ^{35}Cl SP-ICP-MS time scans for PVC food-grade hose migration assays with ultrapure water: (a) 20 °C, 24 h; (b) 20 °C, 72 h; (c) 70 °C, 24 h; (d) 70 °C, 72 h. Red line: critical value for identification of particle events ($\alpha = 2.867 \times 10^{-7}$; 5σ).

events above the threshold value could be distinguished, with 85 particle events being detected, which exceeds the established critical value of 32 events. In order to increase the number of particles detected in the released suspensions at 70 °C for 72 h, acquisition time was extended to 3 min. In addition, dilutions were performed in an attempt to decrease the baseline to improve detection capability. The increase in acquisition time led to the detection of 90 events in ultrapure water, meaning a critical value for particle detection of $Y_{C,N} = 113$ events, which would correspond to a particle number concentration critical value (X_C^{number}) of 1.2×10^6 L^{-1}. Under such conditions, a higher number of events above the threshold value was

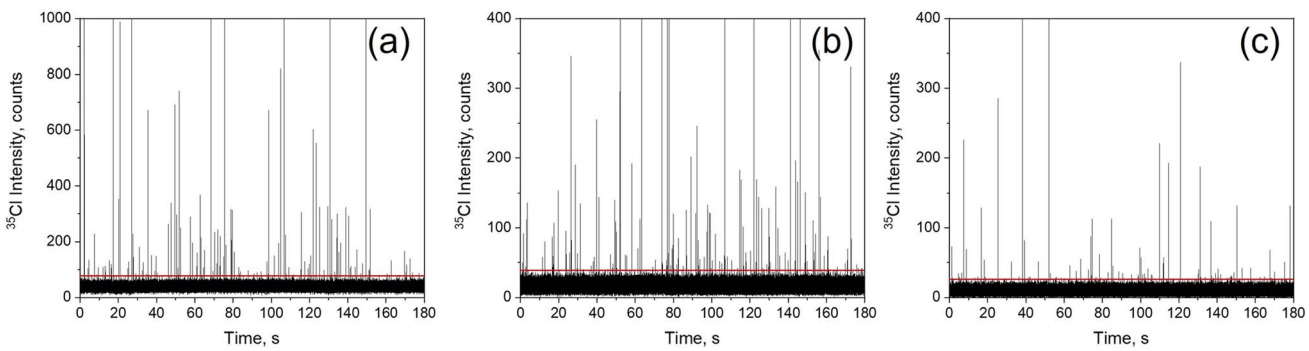

Fig. 4 ^{35}Cl SP-ICP-MS time scans for PVC food-grade hose migration assays at 70 °C for 72 h with ultrapure water with an acquisition time of 3 min: (a) no dilution; (b) 1 : 3 dilution; (c) 1 : 8 dilution. Red line: critical value for identification of particle events ($\alpha = 2.867 \times 10^{-7}$; 5σ).

Table 3 SP-ICP-MS results obtained for PVC food-grade hose migration assays at 70 °C for 72 h with ultrapure water. Mean ± standard deviation ($n = 5$)

Acquisition time	Dilution	Mean baseline intensity, counts	Number of particles	Size range[a], μm
1 min	1 : 1	37.80 ± 0.18	85 ± 5	0.56–2.91 (0.86)
	1 : 3	15.73 ± 0.12	49 ± 6	0.45–2.71 (0.70)
	1 : 8	9.27 ± 0.03	35 ± 8	0.40–2.20 (0.58)
3 min	1 : 1	37.54 ± 0.18	146 ± 43	0.54–3.15 (0.84)
	1 : 3	15. 89 ± 0.12	156 ± 25	0.47–2.11 (0.69)
	1 : 8	9.17 ± 0.03	107 ± 23	0.41–1.80 (0.55)

[a] Median size value in brackets.

observed, as seen in the time scans in Fig. 4 and results summarized in Table 3.

Results in Table 3 show a significant decrease in the baseline levels with the application of successive dilutions of the suspensions, which would be related to the presence of dissolved chlorine species, most probably vinyl chloride monomers and oligomers from the PVC itself, arising from incomplete polymerization during manufacturing or from degradation of the product.[44–46] These species would contribute to the constant baseline observed and their presence would decrease upon dilution. The decrease in the baseline levels leads to a lower size detection threshold and, hence, an improvement in the ability to detect smaller particles. Although this allows the detection of a higher number of particles, it also reduces the particle detection frequency (fewer particles per unit of volume due to the dilution). Equivalent size ranges obtained by SP-ICP-MS, with median values in brackets, are also shown in Table 3. Size detection limit for these experimental conditions was 0.4 μm, ensuring the ability to effectively distinguish between the particles detected and the baseline. Although SP-ICP-MS was able to detect chlorine-containing particles in the PVC food-grade hose migration assays at 70 °C and 72 h, their low concentration hampered the observation of PVC microparticles by electron microscopy, preventing the confirmation of their presence in the leachate by this latter technique.

Likewise, the sample obtained from the migration assay with 3% acetic acid (food simulant) performed for 10 days at 40 °C was also analyzed. Fig. 5 shows the time scans obtained by SP-ICP-MS for the undiluted and diluted samples. The time scan for the 3% acetic acid blank is shown in Fig. S5 for comparative purposes.

In the case of the undiluted sample (Fig. 5a), just 6 events above a baseline >160 counts were detected, which set a particle detection threshold at an intensity of 270 counts. A significant decrease of this baseline was observed upon dilution (Fig. 5b), which reduced the threshold to an intensity of 36 counts, allowing the detection of 9 events in 1 min. However, it should be noted that 44 particles were detected in the 3% acetic acid blank, which established a critical value for particle detection of $Y_{C,\ N} = 60$ events. Therefore, based on the results obtained following the procedure established by Regulation No. 10/2011 of the European Commission, it cannot be guaranteed that particle release above the established detection limit (6×10^6 L^{-1}) will take place. However, it should be noted that an increase in the chlorine baseline levels was observed in

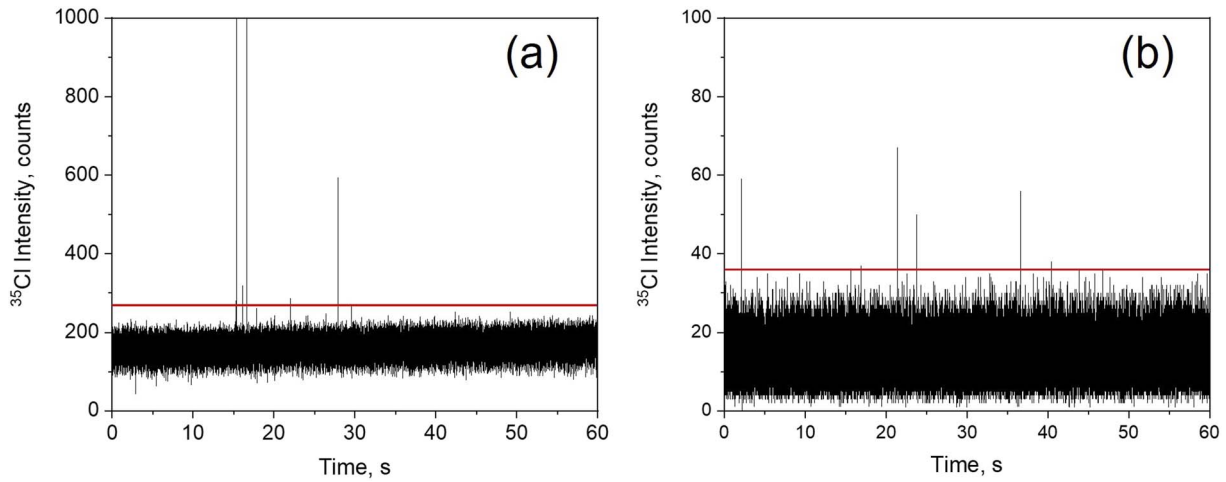

Fig. 5 ^{35}Cl SP-ICP-MS time scans for PVC food-grade hose migration assays with 3% acetic acid for 10 days at 40 °C: (a) no dilution; (b) 1 : 30 dilution. Red line: critical value for identification of particle events ($\alpha = 2.867 \times 10^{-7}$; 5σ).

migration assays in comparison to the corresponding blank (>160 counts *vs.* 7.6 counts), which once again would be related to the presence of vinyl chloride monomers and oligomers released from the original material.

All in all, the results obtained show that the use of PVC materials in the food industry is safe in terms of microplastic release, within the specifications established by the manufacturer. In contrast, its use outside the specified temperature conditions causes a significant release of these microplastics.

3.4.2 Release of PVC microplastics from unplasticized PVC drain pipes. Another common use of PVC is in drain pipes, which are in frequent contact with running water and waste, potentially promoting their degradation and the release of particles into the environment. Therefore, a study focused on the potential release of microplastics from a PVC drain pipe under different operating conditions (20 °C, 40 °C, and 60 °C for 72 h) was performed. Time scans for the different assays are shown in Fig. S6. Results obtained showed that the number of particle events detected was below the critical value calculated for these conditions (48 events) in all the cases. This indicates that, under the assay conditions, PVC particle release into the environment cannot be confirmed. Despite the long contact with water, even at high temperatures above manufacturer recommendations (45 °C), no material degradation leading to the release of microplastics into the environment was observed. These results indicate the stability of PVC used in domestic and industrial applications, although its use under more extreme pressure conditions to assess possible release should be explored.

4. Conclusions

To study the potential effects of microplastics and nanoplastics on the environment and human health, model particles that mimic those found in the environment are needed. In addition to selecting the appropriate protocol for their preparation, the particles must be properly characterized, which can be challenging when dealing with sizes in the micrometre range or smaller. In this regard, an analytical methodology based on SP-ICP-MS has been developed for the detection and size characterization of PVC micro/nanoplastics, which has been complemented with dynamic image analysis to obtain morphological information. A top-down approach was applied based on the cryogenic grinding of the raw material, followed by the fractionation of the particles to the desired size range by centrifugation. The optimized procedure allowed the isolation of a nominal fraction <3 μm. Furthermore, the use of isotopes different to carbon allowed the size detection limits to be reduced below 1 μm, enabling the detection of PVC particles down to 0.35 μm by monitoring ^{35}Cl. The monitoring of this intrinsic element undoubtedly improved SP-ICP-MS capabilities for the detection of nanoplastics. The preparation protocol for micro/nanoplastics is suitable for being scaled up in order to prepare larger amounts of model micro/nanoplastics, which would be characterized using the methodology developed. In addition, the SP-ICP-MS method developed was applied as a screening method for detecting the release of microplastics from industrial PVC products, confirming that there was no apparent degradation of the material in terms of microplastic release according to the manufacturer's specifications, which was not the case at temperatures above those recommended. Further studies concerning aged PVC industrial products, which could be more prone to the release of microplastic particles and other chlorine containing species, as well as migration assays under more severe conditions, should be considered to monitor and assess the risk and behaviour of these materials.

Author contributions

Isabel Abad-Alvaro: formal analysis, investigation, data curation, methodology, conceptualization, supervision, writing – original draft, writing – review & editing. Inés Lázaro-Fernández: formal analysis, investigation, data curation, methodology. Eduardo Bolea: data curation, methodology, conceptualization, supervision, writing – review & editing, funding acquisition. Francisco Laborda: conceptualization, supervision, writing – review & editing, funding acquisition.

Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). The SI contains additional experimental data and extended figures supporting the findings of this study. SI is available online alongside the published article. Supplementary information is available. See DOI: https://doi.org/10.1039/d5ja00455a.

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